Libby Standard Operating Procedure
Approved for Use at the Libby Superfund Site Only
Analysis of Asbestos in Dustfall Samples by TEM (Revision 0)

Date: 05/04/05 SOP No. SRC-LIBBY-07

Title: ANALYSIS OF ASBESTOS IN DUSTFALL SAMPLES BY TEM

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SYNOPSIS: A standardized method is presented for measuring asbestos concentrations in dust released to air during site cleanup activities such as soil removal or building demolition. This method is adapted from methods ASTM D5755-95 and ISO 10312.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8	Peggy Churchill, RPM	5/4/05
Syracuse Research Corp.	W S Britton	5/4/05

Revision	Date	Reason
0	05/04/2005	--

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for transmission electron microscope (TEM) analysis of asbestos in samples of dust collected by a passive fallout collector as described in SOP SRC-LIBBY-06. This procedure is intended for use by employees of USEPA Region 8 and by contractors and subcontractors supporting USEPA Region 8 projects and tasks for the Remedial Investigation work performed at the Libby, Montana, Superfund site.

2.0 RESPONSIBILITIES

The Laboratory Director is responsible for ensuring that fallout samples provided to the laboratory for evaluation are handled and evaluated in accord with the requirements of this SOP, and for communicating to the appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist any recommended changes or proposed improvements to the SOP.

3.0 EQUIPMENT

- Filtered, deionized (FDI) water - sample suspension medium
- Clean 500 mL graduated cylinder
- Filter apparatus, glass or disposable
- 25-mm or 37-mm diameter cellulose ester (MCE) filters with 0.45 um or smaller pore size
- Tweezers - MCE filter preparation for TEM
- Scalpel blade - MCE filter preparation for TEM
- Dimethylformamide/acetic acid mixture - MCE filter preparation for TEM
- Micropipette with disposal tips - MCE filter preparation for TEM
- Plasma etcher -MCE filter preparation for TEM
- Carbon coater - MCE filter preparation for TEM
- Jaffe washer - MCE filter preparation for TEM
- Acetone vapor generator- MCE filter preparation for TEM
- TEM- 80 to 120 kV transmission electron microscope (TEM), capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDS) and it must have a scanning TEM (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover. The microscope must be calibrated and maintained according to the requirements described in NVLAP Airborne Asbestos Program.

4.0 METHOD SUMMARY

Samples of dust fallout will be provided to the laboratory in capped collection cylinders that are about 6 inches in diameter and about 12 inches tall. The cylinders will contain a

layer of water in the bottom to help ensure that all particles which entered the cylinder are retained. At the laboratory, the contents of the cylinder will be collected onto an MCE filter by vacuum filtration. This filter is then prepared and examined for asbestos structures by TEM in basic accord with ISO 10312. The units of the results may be expressed either as total asbestos fallout (s/cm^2) or as a rate of fallout (s/cm^2/hr).

5.0 SAMPLE PREPARATION AND ANALYSIS

5.1 Sample Filtration

Pour the water from the collection cylinder into a clean 500-mL graduated cylinder. Rinse the container thoroughly with FDI water and collect the rinsate in the graduated cylinder. Add additional FDI water to a final volume of 500 mL. Thoroughly mix the 500 mL sample by hand-inverting 10 times.

Remove 250 mL of the sample suspension and filter through a 25 mm or 37 mm MCE filter (0.45 um or smaller pore size) using a disposable filter funnel. If the dust loading on the filter is too heavy, prepare a second filter using a smaller volume.

5.2 TEM Filter Preparation

Prepare at least two grids from the filter for examination by TEM in accord with the standard methods described in International Organization for Standardization (ISO) method 10312, except where specifically indicated in this method or where appropriate project-specific laboratory modifications are necessary.

5.3 Counting Rules

Counting rules are the same as described in ISO 10312, except that all asbestos structures 0.5 um in length and with an aspect ratio of at least 3:1 should be recorded. Target sensitivity and appropriate stopping rules should be specified in the workplan or QAPP/SAP developed for the project.

6.0 COMPUTATION OF RESULTS

The amount of asbestos in dust fallout during a sampling period is calculated using the following equation:

$$AFO = \frac{N \cdot EFA}{GO \cdot Ago \cdot A \cdot F}$$

where:

AFO = Asbestos fallout (structures / cm^2)
N = Number of countable asbestos structures observed
EFA = Effective filter area (mm^2)

GO = Number of grid openings examined
Ago = Area of one grid opening (mm^2)
A = Area of collection cylinder (cm^2)
F = Fraction of original sample applied to filter

If the number of asbestos structures observed is zero, the results should be reported as less than the analytical sensitivity , where sensitivity is given by:

$$S = \frac{EFA}{GO \cdot Ago \cdot A \cdot F}$$

For convenience, all of these calculations are performed automatically by the electronic analytical data recording sheet (Attachment 1).

Asbestos fallout rate (AFR) is computed from AFO as follows:

AFR = AFO / Collection time (hr)

7.0 QUALITY ASSURANCE

Laboratory blanks should be prepared and analyzed in accord with standard laboratory practice. If asbestos contamination is detected on a laboratory blank sample, the laboratory director should take immediate steps to identify and address the source of the contamination before any further field samples are analyzed.

8.0 DOCUMENTATION

All analytical results for each sample should be recorded using the standard electronic data sheet provided in Attachment1.

9.0 REFERENCES

American Society for Testing and Materials. 1995. Standard Test method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations. ASTM Method D 5755-95.

International Organization for Standardization. 1995. Ambient air -- Determination of Asbestos Fibres -- Direct Transfer Transmission Electron Microscopy Method. ISO Method 10312.

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ATTACHMENT 1

Electronic Datasheet for Recording Analytical Results

TEM.xls
(Check with Volpe or SRC to determine the most recent version number)